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KOH etching of (100) Si wafer, No 1

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KOH etching Silicon

Disciplines
Biological and Chemical Physics | Engineering Physics | Inorganic Chemistry | Materials Chemistry | Membrane Science | Other Chemical Engineering | Other Materials Science and Engineering | Physical Chemistry | Structural Materials

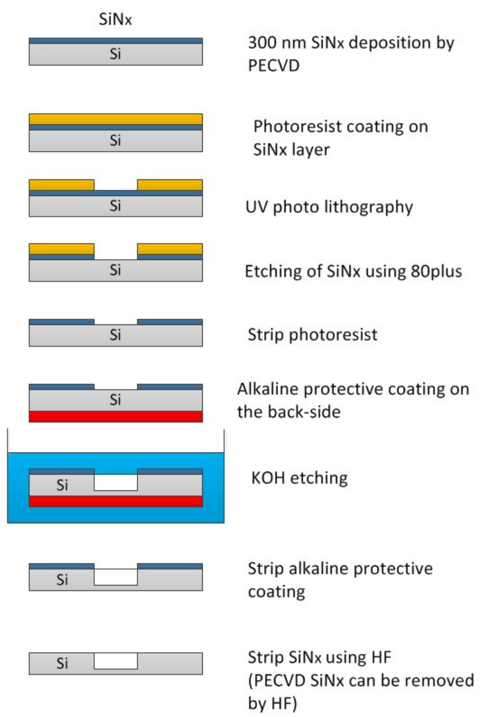


Figure 1. Process flow of KOH etching of (100) Si wafer

Table 1. PECVD deposition parameters of default and Test recipes.

	Default	Test
Set Pressure (Torr)	1.0	1.8
Temperature (°C)	350	350
LF Power		
Forward (W)	20	160
Pulse Time (sec)	7	8
Pulsed	ON	ON
LF First	OFF	OFF
RF Power		
Forward (W)	20	200
Pulse Time (sec)	13	12
Pulsed	ON	ON
HF First	ON	ON
RF Automatch		
Capacitor 1	77	77
Capacitor 2	26	26
Auto/Manual/Hold	Hold	Hold
Gas flow (sccm)		
Silane (90% He)	170	90
NH3	20	45
N2	820	1305

1. Introduction

The goal of this project is to perform on-site inspection of potassium hydroxide (KOH) wet etching process, using the tools available at Quattrone Nanofabrication Facility. Figure 1 shows a process flow of KOH etching of (100) Si wafer. A 300 nm thick silicon nitride film was used as a hard mask against KOH etching.

2. Experimental Section

A. Deposition of 300 nm thick silicon nitride film

A (100) Si wafer was sonicated in acetone and isopropyl alcohol (IPA) for 5 min each, and was dried using nitrogen gas blow. A 300 nm thick silicon nitride film was deposited on the Si wafer for a hard mask upon KOH etching, using Oxford Plasma Lab 100 (Plasma Enhanced Chemical Vapor Deposition (PECVD)).

Table 1 indicates deposition parameters of the default and Test recipes. The default recipe was given by Oxford Instruments. The deposition time was 27 min and 16 sec. However, the silicon nitride film prepared by the default recipe did not work as a hard mask; it was easily etched through into Si upon KOH etching, and the resultant surface had uneven cavities all over. On the other hand, the silicon nitride film prepared by the "Test" recipe worked as the hard mask, and was also easily removed using hydrofluoric acid (HF) solution, as described later. The deposition time was 6 min and 30 sec.

B. UV lithography using SUSS MicroTec MA6 Gen3 Mask Aligner

Hexamethyldisilazane (HMDS) was vapor primed on the 300 nm thick silicon nitride film as an adhesion promoter, using YES oven (Yield Engineering Systems), followed by spin-coating positive photoresist S1818 (Microchem) at 5500 rpm for 30 sec. The photoresist film was baked at 115^{o}C for 5 min on a hot plate, and was exposed to 405 nm UV light with the power of 150mJ/cm^2, using SUSS MicroTec MA6 Gen3 Mask Aligner. The exposed photoresist film was developed in MF319 (Microchem) for 1 min and 30 sec, and was rinsed with deionized (DI) water. The sample was then dried using nitrogen gas blow.

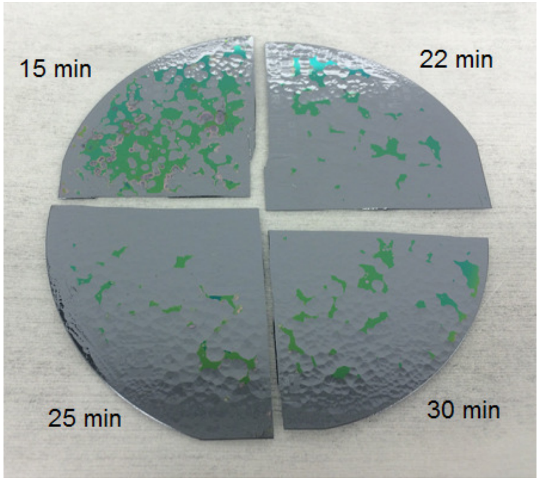


Figure 2. A photo image of four pieces of the Si wafer with 300 nm thick silicon nitride film, prepared by the default recipe, on its surface. The four pieces were immersed into 30 wt% KOH solution at 80 °C for 15, 22, 25, and 30 min, respectively.

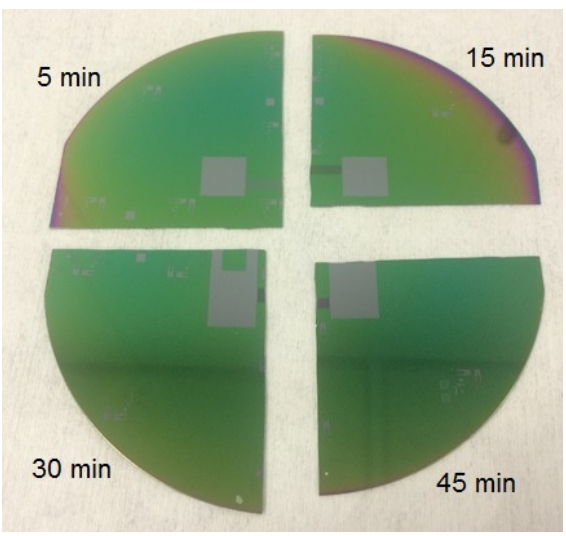


Figure 3. A photo image of four pieces of the Si wafer with 300 nm thick silicon nitride film, prepared by the Test recipe, on its surface. The four pieces were immersed into 30 wt% KOH solution at 80 °C for 5, 15, 30, and 45 min, respectively.

C. <u>Dry etching of silicon nitride layer using Oxford 80 plus RIE</u>

The silicon nitride film was dry-etched through the developed photoresist film, using Oxford 80 plus RIE with the following condition (the default recipe): O_2 = 5 sccm; CHF_3 = 50 sccm; pressure = 20 mTorr; power = 150 W; T = 17.5 °C. The etch rate was 61 nm/min. After the etching, the photoresist film was removed by sonication in Remover PG (Microchem) at 60 ^{o}C.

D. <u>Alkaline protective coating on the back side of the Si wafer</u>

ProTEK B3 Primer (Brewer Science) was spin coated on the back side of the Si wafer at 1500 rpm for 60sec, and was baked at 205 ^{o}C for 60 sec on a hot plate, as an adhesion promoter. Then, ProTEK B3 Protective Coating (Brewer Science) was spin coated on the primer at 1000 rpm at 60 sec, and was baked at 120 ^{o}C for 120 sec on a hot plate, followed by the 2nd baking at 205 ^{o}C for 60 sec.

E. <u>KOH etching</u>

Figure 2 shows a photo image of four Si pieces with 300 nm thick silicon nitride film, prepared by the default recipe, on its surface. The four pieces were immersed into 30 wt% KOH aqueous solution at 80 °C for 15, 22, 25, and 30 min, respectively. As can be seen in figure 2, the silicon nitride film (refractive index, n = 1.98-2.03 at 632.8 nm) prepared by the default recipe was easily etched through into Si upon KOH etching, and the surface had uneven cavities all over.

Figure 3 shows a photo image of four Si pieces with 300 nm thick silicon nitride film, prepared by the Test recipe, on its surface. The four pieces were immersed into 30 wt% KOH aqueous solution at 80 °C for 5, 15, 30, and 45 min, respectively. As can be seen in figure 3, the silicon nitride film (n = 1.86 at 640 nm) prepared by the Test recipe was not damaged at all against KOH solution.

The etch rate of PECVD silicon nitride in 30 wt% KOH solution at 80 °C is reported to be 0.67 nm/min for the film with low refractive index, whereas it is 0 nm/min for the film with high refractive index (J. Microelectromech. Syst. 12, 761 (2003)). This strongly suggests that stoichiometry Si_3N_4 of the silicon nitride film prepared by the default recipe should not be uniform, although the refractive index of 1.98-2.03 at 632.8 nm is almost the same as the literature value of 2.01 at 632.8 nm (http://refractiveindex.info/?shelf=main&book=Si3N4&page=Philipp).

F. Strip alkaline protective coating

It was very difficult to remove the alkaline protective coating film using ProTEK Remover 100 (Brewer Science). However, the alkaline protective coating film could easily be peeled off during the process of stripping the silicon nitride film in 49wt% HF aqueous solution.

G. Strip silicon nitride layer

Etchants of PECVD silicon nitride have been known to be a 5:1 mixture of 40wt% NH_4F and 49wt% HF and 85wt% phosphoric acid (J. Microelectromech. Syst. 12, 761 (2003)). On the other hand, etching of Low-Pressure Chemical Vapor Deposition (LPCVD) silicon nitride using a 10:1 mixture of H_2O and 49wt% HF has been shown to be slow or zero, but etching of PECVD silicon nitride using HF solution has not been reported. In this report, PECVD silicon nitride prepared by the Test recipe was easily removed using 49wt% HF solution. This suggests that the stoichiometry of the silicon nitride film should be similar to that of LPCVD silicon nitride. Further investigation is needed to elucidate the relationship between the etching property and the stoichiometry.

3. Results

Figures 4 to 7 show SEM images of 20 µm width opened and 20 µm width masked lines, 2.4 µm width opened and 3.6 µm width masked lines, 5 µm width right angle lines, and 82.5 µm x 82.5 µm squares, respectively. The etch rate along the (100) direction was determined to be 0.9-1.5 µm/min, which was scattered from the literature value =1.3-1.4 µm/min (for the (100) direction in 30wt% KOH solution at 80 ^{o}C) because the KOH solution was not stirred during the etching.

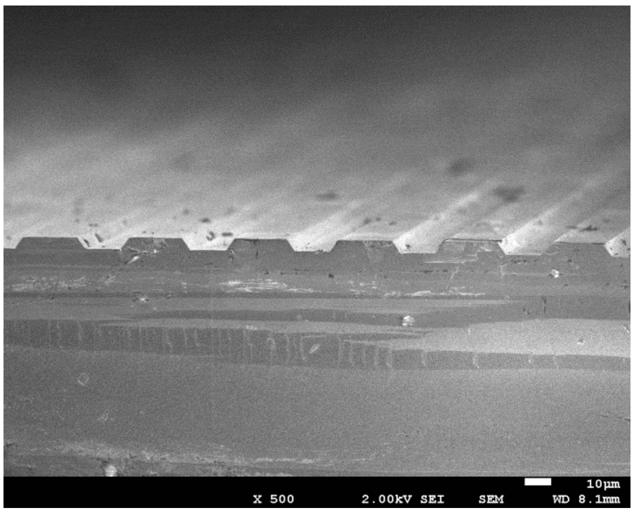
The SEM images shown in figures 4 to 7 also indicate that the KOH etching is not perpendicular to the (100) crystal plane, but forms the pyramidal structures. It is known that a (100)-orientated wafer forms square-based pyramids with (111) crystal planes because the bonding energy between Si atoms depends on the crystal planes, resulting in the highly anisotropic etching. Figure 5 indicates that the grating structures can be formed until ~3 µm width and ~3 µm depth at least. In addition, figure 7 shows pyramidal etching in the opened squares. On the other hand, the undercut beneath the hard mask is also observed due to the highly anisotropic etching, as shown in figures 4 and 5.

4. Summary

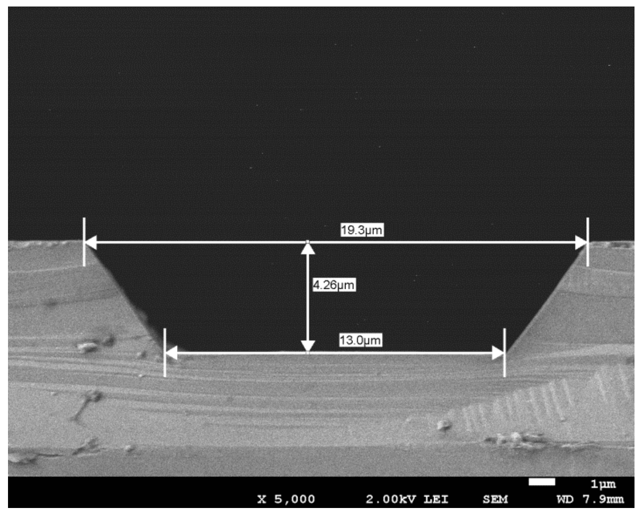
KOH wet etching of (100) Si wafer was performed, using the tools available at Quattrone Nanofabrication Facility. A 300 nm thick silicon nitride film was used as the hard mask against KOH etching. However, the PECVD silicon nitride film prepared by the default recipe given by Oxford Instruments was easily etched through into Si upon KOH etching, and the resultant surface had uneven cavities all over, although the refractive index was almost the same as the literature value. On the other hand, the silicon nitride film prepared by the “Test” recipe worked as the hard mask against KOH etching, and furthermore was easily removed using HF solution.

Further investigation is needed to elucidate the relationship between the etching property and the stoichiometry.

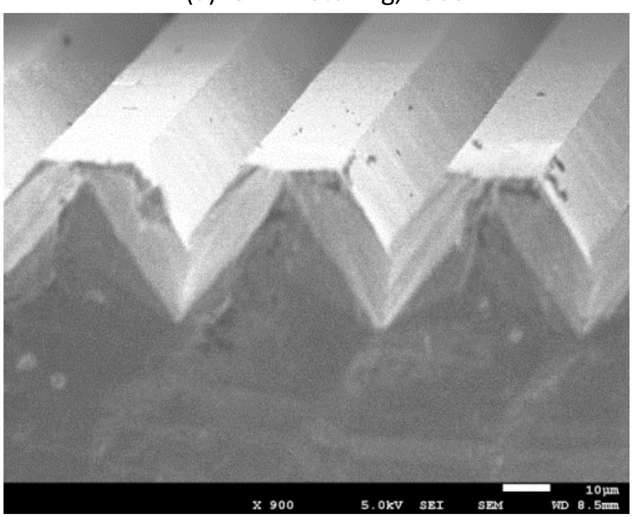
The etch rate along the (100) direction was determined to be 0.9-1.5 µm/min. The KOH etching along the (100) direction formed the pyramidal structures with (111) crystal planes, due to the highly anisotropic etching. The line etching created the grating structure with ~3 µm width and ~3 µm depth, and the square etching formed a pyramidal structure. On the other hand, the undercut beneath the hard mask was also observed due to the highly anisotropic etching.



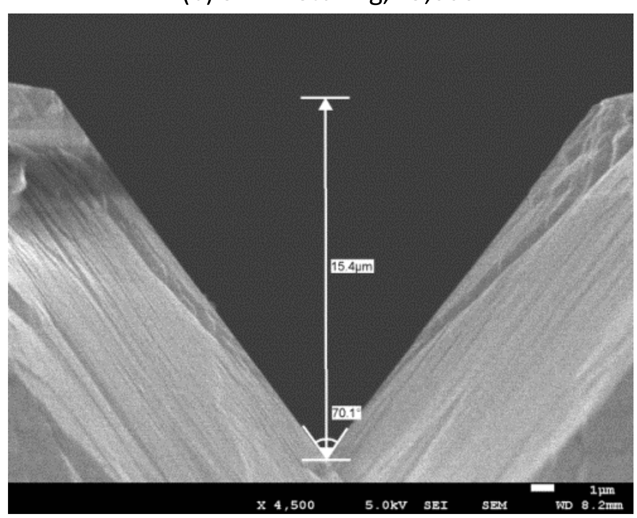
(a) 5 min etching, x500



(b) 5 min etching, x5,000

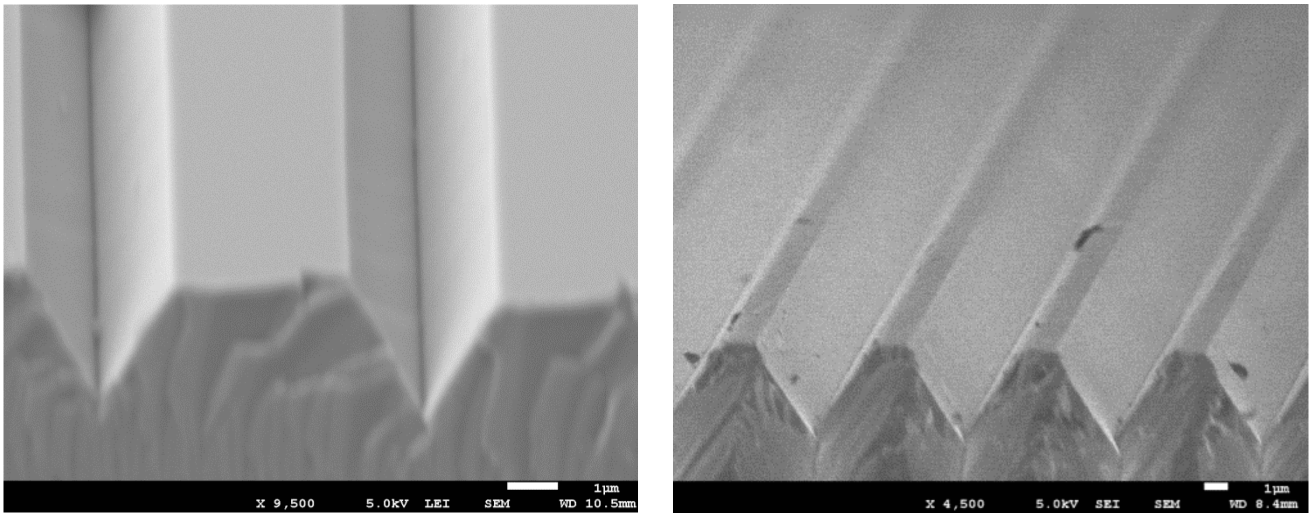


(c) 30 min etching, x900



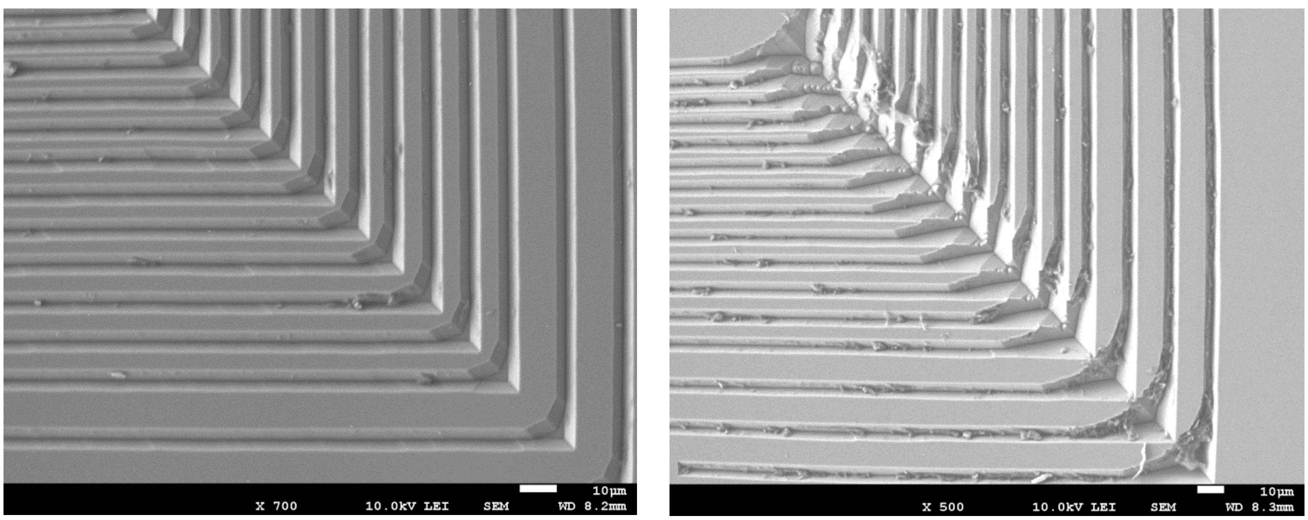
(d) 30 min etching, x4,500

Figure 4. SEM images of cross-sections of 20 µm width opened and 20 µm width masked lines etched for 5 and 30 min. (a) 5 min etching, x500; (b) 5 min etching, x5,000; (c) 30 min etching, x900; (d) 30 min etching, x4,500. The etch rate is 0.9 µm/min.



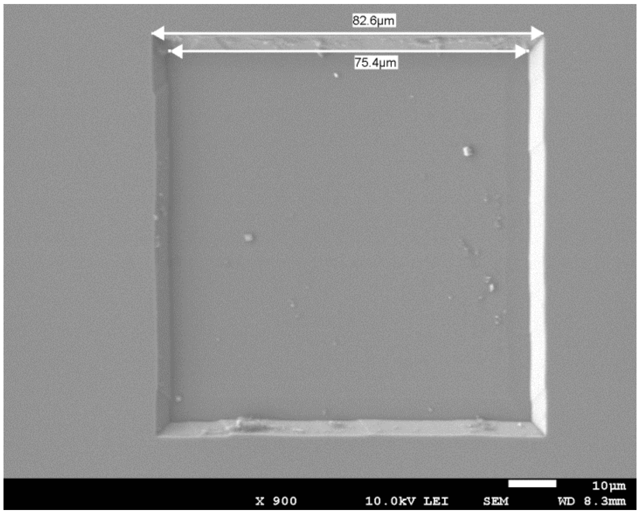
(a) 15 min etching, x9,500 (b) 45 min etching, x4,500

Figure 5. SEM images of cross-sections of 2.4 µm width opened and 3.6 µm width masked lines etched for 15 and 45 min. (a) 15 min etching, x4,500; (b) 45 min etching, x4,500.

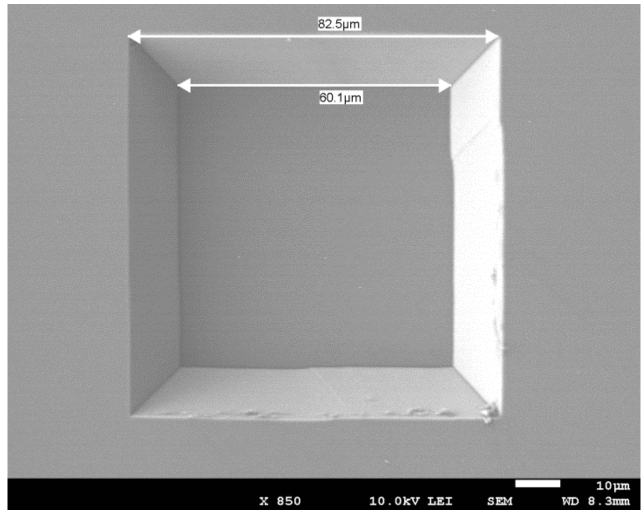


(a) 5 min etching, x700 (b) 15 min etching, x500

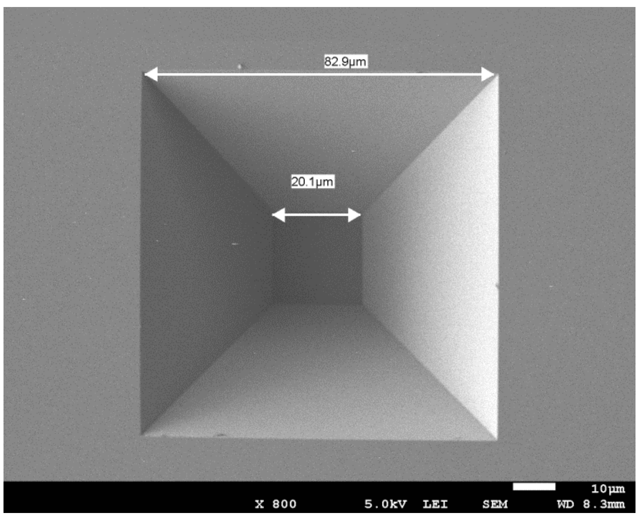
Figure 6. SEM images of 5 µm width right angle lines. (a) 5 min etching, x700; (b) 15 min etching, x500.



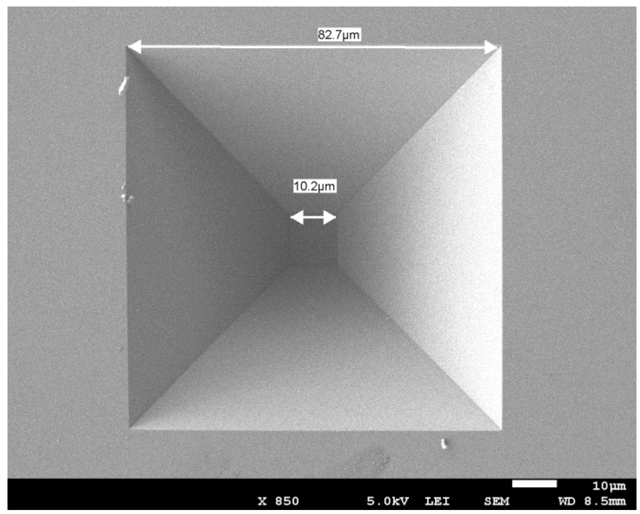
(a) 5 min etching



(b) 15 min etching



(c) 30 min etching



(d) 45 min etching

Figure 7. SEM images of 82.5 µm x 82.5 µm squares etched for (a) 5 min at the etch rate = 1.0 µm/min; (b) 15 min, 1.1 µm/min; (c) 30 min, 1.5 µm/min; (d) 45 min, 1.1 µm/min.